

CLAIMS

1. A heat treatment process in a chemical synthesis, of the dielectric type, characterized in that said dielectric heating is carried out intermittently, that is to say, the reagent or reagents is/are subjected to electromagnetic waves intermittently, in combination with a recycling system.
2. A process as claimed in Claim 1 wherein the electromagnetic waves are selected in the frequencies ranging between 300 GHz and 3 MHz.
3. A process as claimed in Claim 2 wherein the frequencies are selected from among:
 - *MW frequencies and HF*
 - Those microwave frequencies (MW) that range from about 300 MHz to about 30 GHz, preferably standing at 915 MHz (authorized frequency with a tolerance of 1.4%) or at 2.45 GHz (authorized frequency with a tolerance of 2%).
 - Those high frequencies (HF) that range from about 3 MHz to about 300 MHz, preferably standing at 13.56 MHz (authorized frequency with a tolerance of 0.05%) or at 27.12 MHz (authorized frequency with a tolerance of 0.6%).
4. A process as claimed in any of Claims 1 to 3, wherein the entire reaction volume is not continuously exposed to dielectric waves but wherein all reaction mixture molecules are intermittently subjected to the field.
5. A process as claimed in any of Claims 1 to 4, wherein the reagent(s) can be selected from among those products that hardly absorb the electromagnetic waves or those products that are highly absorbent of said waves or a mixture of both, whether or not

enhanced with one or several hardly or highly absorbent catalysts or additives and/or process gas.

6. A process as claimed in Claim 5 wherein the reagent(s) is/are selected from among:

- *vegetable oils*

rapeseed oil, sunflower oil, peanut oil, olive oil, walnut oil, corn oil, soy oil, linseed oil, safflower oil, apricot kernel oil, sweet almond oil, hemp oil, grassed oil, copra oil, palm oil, cottonseed oil, Babes oil, jujube oil, sesame oil, argon oil, milk-thistle oil, gourds oil, raspberry oil, Carnage oil, enema oil, poppies oil, Brazilnut oil, castor oil, dehydrated castor oil, hazelnut oil, wheat germ oil, borage oil, oenothera- oil, Tung oil, or tall oil.

- *animal fats or oils*

sperm-whale oil, dolphin oil, whale oil, seal oil, sardine oil, herring oil, shark (dog-fish) oil, cod-liver oil, neatsfoot oil, as well as beef, pork, horse, mutton tallow (marrow).

- *compounds of animal or vegetable oils*

squalene extracted from non-saponifiable fats of vegetable oils (olive oil, peanut oil, rapeseed oil, corn germ oil, cottonseed oil, linseed oil, wheat germ oil, rice bran oil) or squalene contained in large amounts in shark (dog-fish) oil.

- *hydrocarbons*

unsaturated: alone or in a mixture, an alcene, for example one or several terpenic hydrocarbons, that is to say, one or several polymers of isoprene, or one or several polymers of isobutene, of styrene, of ethylene, of butadiene, of isoprene, of propene, or one or several copolymers of these alcenes.

saturated: alkanes, for example ethane, propane.

- saturated and/or unsaturated esters

alone or in a mixture, one or several esters obtained by esterification between a monoalcohol and/or polyol and at least one saturated and/or unsaturated fatty acid; waxes; butters, phospholipids; spingolipids; glucolipids.

- saturated and/or unsaturated acids

alone or in a mixture, one or several saturated acids such as caprylic acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, behenic acid, lignoceric acid, cerotic acid, one or several monounsaturated fatty acids such as oleic acid, palmitoleic acid, myristic acid, petroselenic acid, erucic acid; one or several polyunsaturated fatty acids such as for example linoleic acid, alpha and gamma linolenic acids, arachidonic acid; 5c,8c,11c,14c-eicosapentaenoic acid (EPA), 4c,7c,10c,13c,16c,19c-docosahexaenoic acid (DHA), one or several acids comprising conjugated dienes or conjugated trienes such as licanic acid, isomers of linoleic and linolenic acids; one or several acids comprising one or several hydroxyl groups such as ricinoleic acid.

- *alcohols*

glycerol, sorbitol, sucrose, mannitol, xylitol, neopentylglycol, pentaerythritol, saccharose, galactose, glucose, maltose, maltotriose, fructose, maltitol, lactitol, lactose, ribose, mellibiose, cellobiose, gentiobiose, altrose, gulose, polyalkyleneglycols, polyglycerols, polyphenols, alkylpolyglucosides, polyglucosides, glycol, pentaerythritol, 1,2-ethanediol, 1,4-butanediol; 1,6-hexanediol, aminoalcohols (for example, diethanol amine (DEA), triethanol amine (TEA), 3-amino-1,2-propanediol), epoxyalcohols, saturated or unsaturated fatty alcohols (for example, myristyl alcohol, oleyl alcohol, lauryl alcohol),

linear or branched alcohols, vitamins (for example, tocopherol, ascorbic acid, retinol), sterols (including phytosterols), hemiacetals (for example, 1-ethoxy-1-ethanol), aminoalcohols (for example, 2-2'-aminoethoxy ethanol), epoxyalcohols (for example, 2-3-epoxy-1-propanol), propanol, ethanol, methanol, tetradecyl alcohol and their analogs.

- *epoxides*

alone or in a mixture, vernolic acid, coronaric acid, 1,2-epoxy-9-decene, 3-4-epoxy-1-butene, 2-3-epoxy-1-propanol, fatty esters obtained by esterification between 2-3-epoxy-1-propanol and a fatty acid (for example, Cardura E10®).

- *amino alcohols*

alone or in a mixture, monoethanol amine (MEA), diethanol amine (DEA), triethanol amine (TEA), 3-amino-1,2-propanediol, 1-amino-2-propanol; 2-2'-aminoethoxy ethanol.

- *amines*

ammonia, primary, secondary and tertiary alkyl amines (for example, methyl amine, dimethyl amine, trimethyl amine, diethyl amine), fatty amines (for example, oleic amines, coconut alkyl amines), amino alcohols (for example, monoethanol amine (MEA), diethanol amine (DEA), triethanol amine (TEA), 3-amino-1,2-propanediol, 1-amino-2-propanol), ethoxylated amines (2-2'-aminoethoxy ethanol, amino-1-methoxy-3-propane), which amines can be saturated or unsaturated, linear or branched.

7. A process as claimed in Claim 6, wherein the animal or vegetable fats and oils, as well as their derivatives, can undergo a prior treatment intended to make them on the one hand more reactive or on the other hand less reactive (both an isolated reagent and a reaction mixture comprising two or several compounds, which reaction mixtures can

comprise equivalent proportions of each compound, or some compounds can be majority compounds).

8. A process as claimed in Claim 6 or 7, wherein the alcohols as well as their derivatives can undergo a prior treatment intended to make them on the one hand more reactive or on the other hand less reactive, such as for example hydrogenation, hydroxylation, epoxidation, phosphitation, sulfonation.

9. A process as claimed in any of Claims 1 to 8, wherein it uses as catalysts or additives:

- catalysts

the common acid catalysts (para toluene sulfonic acid, sulfuric acid, phosphoric acid, perchloric acid, etc.), the common basic catalysts (soda, potash, alcoholate of alkaline metals and of alkaline-earth metals, sodium acetate, triethyl amines, pyridine derivatives, etc.), acid and/or basic resins of the Amberlite™, Amberlyst™, Purolite™, Dowex™, Lewatit™ types, zeolithes and enzymes, carbon blacks, and activated carbon fibers.

10. A process as claimed in any of Claims 1 to 9, wherein the volume exposed to electromagnetic waves is with:

- one (1) 6kW magnetron generator operating at the 24500 MHz frequency for the laboratory treatments
- one (1) 60kW magnetron generator operating at the 915 MHz frequency for the industrial treatments

	D (mm)	H (mm)	Unit Vexp	Number of reactors	Total Vexp
Pilot	30	45	32 mL	1	32 mL
Industrial	100	124	1 L	4	4 L

wherein:

D = diameter of cylindrical reactors = 2R

H = height of waveguide

Unit Vexp = volume exposed to waves on a continuous basis for one reactor

Total Vexp = volume exposed to waves on a continuous basis for both reactors

$$V \text{ (exposed to the field)} = \Pi * R^2 * H$$

11. A device for the implementation of the process as claimed in any of Claims 1 to 10, wherein it comprises or consists of :

A)

- pumps
- reactors subjected to the electromagnetic field
- a dielectric system: chimney applicators, generator waveguides, iris, short-circuit piston, cooling systems
- buffer reactors
- tanks
- a gas circuit, preferably for an inert gas such as nitrogen
- condensers
- measuring devices

-and in particular B)

- pumps

The pump(s) is/are of the variable flow type.

It can be a feeder dosing pump and/or a recycling pump and/or a vacuum pump. The outflow of the recycling pump influences the time required for a molecule to transit under the waves.

The pumps can be selected, for purpose of indication, from among vane pumps or piston pumps.

- one or several reactors subjected to electromagnetic waves

a) The reactors subjected to the electromagnetic field do not absorb waves (pyrex, quartz, etc.).

b) They are typically cylindrical in shape.

c) They are positioned inside the applicators.

- a dielectric system: energy applicators, chimneys, waveguides, generator, iris, and short-circuit piston, cooling systems.

- a) The applicators are formed by singlemode cavities that resonate at the transmission frequency according to a radiation in the direction of the waveguide.
- b) The chimneys prevent wave leakage to the outside of the waveguide. They are preferably of a conical cylindrical shape, as indicated in Application FR N° 0108906 filed by this Applicant for limiting the presence of electric arcs.
- c) The waveguide(s) carries/carry the electromagnetic waves. Each waveguide can be subdivided into two — and only two — waveguides.
- d) The generators used are microwave or high-frequency generators.
- e) The microwave (MW) frequencies range from about 300 MHz to about 30 GHz, preferably standing at 915 MHz (authorized frequency with a tolerance of 1.4%) or at 2.45 GHz (authorized frequency with a tolerance of 2%).
- f) The high frequencies (HF) range from about 3 MHz to about 300 MHz, preferably standing at 13.56 MHz (authorized frequency with a tolerance of 0.05%) or at 27.12 MHz (authorized frequency with a tolerance of 0.6%).
- g) The generators are outfitted with a safety feature that allows the incident waves to pass through and that diverts the reflected waves to a water load in which the waves are absorbed.
- h) These generators also require the use of iris, of a shortcircuit piston in order to decrease the reflected power and to promote absorption of the generator-transmitted power by the reaction mixture.
- i) The system is outfitted with cooling systems in order to avoid any overheating.

- buffer reactors

The buffer reactors permit treating a larger amount of reaction mixture.

- tanks

The system is outfitted with one or several feeder tank(s), receiver tank(s), filtration tank(s).

- gas circuits

The heat treatments are carried out under a normal atmosphere, or an oxygen-rich atmosphere or, preferably, an inert atmosphere.

- measuring devices

The system is outfitted with measuring devices such as manometers, thermocouples, flowmeters.

12. A device as claimed in Claim 11, wherein the process can be used in dynamics or in continuum.

13. A device as claimed in Claims 11 or 12, wherein it uses as energy applicator:

- *type of energy applicators*

— As regards high frequency applicators, they consist mainly in:

- applicators of the capacitive type formed by two condenser armatures between which the generator's high-frequency voltage is applied. They are used for the

heat treatment of materials whose volume constitutes a parallelepiped, one side of which is sufficiently thick (>10 mm).

- applicators with rods for planar materials. These applicators are made up of tubular or rod-shaped electrodes. They are used for the heat treatment of materials whose volume constitutes a parallelepiped, one side of which is insufficiently thick (<10mm).

- applicators for filiform materials, formed by loops.

— As regards microwave applicators, we can cite:

- the localized-field applicators: singlemode cavity
- the diffuse-field applicators: multimode cavity
- the near-field applicators: {wave}guide with radiating antennas

14. A device as claimed in any of Claims 11 to 13, wherein :

- The "singlemode" system (localized field) which is formed by singlemode cavities resonating at the transmission frequency according to a radiation in the direction of the {wave}guide, is preferable to the "multimode" (diffuse field).
- The applicator is outfitted with regular cylindrical chimneys.
- The device includes good venting with humid air or with some other comparable gas as regards its dielectric constants (for example, sulfur hexafluoride SF₆ under 1 bar) or chimneys with specially adapted shapes so as to eliminate static electricity formed on the outside wall of the reactor.

15. A device as claimed in any of Claims 11 to 14, wherein a specific chimney geometry is used, in particular a conical chimney.

16. A device as claimed in any of Claims 11 to 15, wherein the reactor is typically cylindrical in shape, and its diameter may not exceed the width of the waveguide.

17. A device as claimed in any of Claims 11 to 16, wherein in the case of singlemode microwave applicators, under 2450 MHz, the waveguide width recommended in order to remain in the TE 0.1 (Transverse Electric) mode stands between about 70 and 100 mm, and more specifically at 90 mm.

18. A device as claimed in any of Claims 11 to 16, wherein in the case of singlemode microwave applicators, under 915 MHz, the waveguide width recommended to remain in the TE 0.1 (Transverse Electric) mode is about 250 mm.

19. A device as claimed in any of Claims 11 to 18, wherein the dielectric system comprises applicators, chimneys, waveguides, a generator, iris, a shortcircuit piston, cooling systems, as follows:

- The applicators are formed by singlemode cavities that resonate at the transmission frequency according to a radiation in the direction of the waveguide.
- The chimneys prevent wave leakage to the outside of the waveguide. They are preferably of a conical cylindrical shape, as indicated in Patent Application FR N°0108906 filed by this Applicant for limiting the presence of electric arcs.
- The waveguide(s) carries/carry the electromagnetic waves. Each waveguide can be subdivided into two — and only two — waveguides.

- The device also uses iris, short-circuit piston in order to lower the reflected power and to promote absorption of the generated-transmitted power by the reaction mixture.

20. A Process and Device as claimed in any of Claims 1 to 19, characterized in that the heat treatments are carried out under a normal atmosphere, or an oxygen-rich atmosphere, or preferably under an inert atmosphere.

21. Applications of the process as claimed in any of Claims 1 to 10 and 20 and of the device as claimed in any of Claims 11 to 19 in all "heat applications", that is to say, those chemical syntheses involving a heat treatment and the use of a single reagent, or a mixture of reagents, in variable proportions, with or without catalysts, with or without process gas.

22. Applications as claimed in Claim 21, wherein as "heat applications" we can cite, as non-limitative examples, such reactions as esterification, transesterification, epoxidation, sulfatation, phosphitation, hydrogenation, peroxidation, isomerization, dehydration, quaternization, amidation, polymerization, polycondensation, and all the common treatments such as decolorizing, deodorizing, and the other systems for eliminating volatile compounds.

23. Applications of the process as claimed by any of Claims 1 to 10 and 21 and of the device as claimed by any of Claims 11 to 19 to all "lipochemistry" reactions.

24. Applications of the process as claimed in any of claims 1 to 10 and 21 and of the device as claimed in any of Claims 11 to 19 for manufacturing polymers of unsaturated fatty acids, of unsaturated fatty acid esters, of unsaturated hydrocarbons or of derivatives of these products using intermittent dielectric heating under microwaves.

25. Applications of the process as claimed in any of Claims 1 to 10 and 21 and of the device as claimed in any of Claims 11 to 19 specific to the synthesis of:

- polyglycerol
- polyglycerol esters
- polyglycerol-6 dioleate
- polyglycerol-2 tristearate